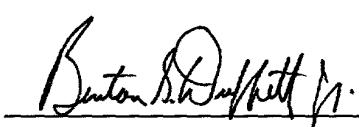


|   |  |   |  |   |
|---|--|---|--|---|
| FORM-PTO-1390<br>(Rev. 12-29-99)  |  | U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE |  | ATTORNEY'S DOCKET NUMBER  |
| <b>TRANSMITTAL LETTER TO THE UNITED STATES<br/>DESIGNATED/ELECTED OFFICE (DO/EO/US)<br/>CONCERNING A FILING UNDER 35 U.S.C. 371</b>   |  |   |  | 003300-852  |
| INTERNATIONAL APPLICATION NO.<br>PCT/SE00/00597   |  | INTERNATIONAL FILING DATE<br>28 March 2000              |  | U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)<br>unassigned <b>09/937851</b> |
| TITLE OF INVENTION<br><b>A PROCESS FOR STERILIZING A BIOLOGICALLY CONTAMINATED ENCLOSURE</b>  |  |   |  |   |
| APPLICANT(S) FOR DO/EO/US<br><b>SHOAA ABDUL RAHMAN and RAÁFAT KITTANEH</b>  |  |   |  |   |
| Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:   |  |   |  |   |
| <ol style="list-style-type: none"> <li><input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li><input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li><input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).</li> <li><input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</li> <li><input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))           <ol style="list-style-type: none"> <li><input checked="" type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li> <li><input checked="" type="checkbox"/> has been transmitted by the International Bureau.</li> <li><input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US)</li> </ol> </li> <li><input type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).</li> <li><input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))           <ol style="list-style-type: none"> <li><input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</li> <li><input type="checkbox"/> have been transmitted by the International Bureau.</li> <li><input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li><input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li><input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li> <li><input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (Signed Declaration will follow).</li> <li><input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</li> </ol> |  |   |  |   |
| <b>Items 11. to 16. below concern other document(s) or information included:</b>  |  |   |  |   |
| <ol style="list-style-type: none"> <li><input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li> <li><input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li> <li><input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment.           <ol style="list-style-type: none"> <li><input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</li> </ol> </li> <li><input type="checkbox"/> A substitute specification.</li> <li><input type="checkbox"/> A change of power of attorney and/or address letter.</li> <li><input checked="" type="checkbox"/> Other items or information:</li> </ol>   |  |   |  |   |
| A certified copy of Swedish Application No. 9901137-1, filed 29 March 1999, was submitted during the international phase of the examination. Thus, the claim for priority has been perfected.   |  |   |  |   |

|   |   |   |
|---|---|---|
| U.S. APPLICATION NO. (If known) (37 CFR 1.492(e))<br>unassigned   | INTERNATIONAL APPLICATION NO.<br>PCT/SE00/00597 | ATTORNEY'S DOCKET NUMBER<br>003300-852                        |
| 17. <input checked="" type="checkbox"/> The following fees are submitted:   |   | CALCULATIONS  |
| <b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b>   |   | PTO USE ONLY  |
| Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1,000.00 (960)                  |   |   |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$860.00 (970)  |   |   |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$710.00 (958)   |   |   |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$690.00 (956)  |   |   |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00 (962)  |   |   |
| <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>   |   | \$ 1,000.00   |
| Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)).   |   | 20 <input type="checkbox"/> 30 <input type="checkbox"/> \$ -- |
| Claims  | Number Filed                                    | Number Extra  |
| Total Claims  | 20 -20 =  | 0   |
| Independent Claims  | 1 -3 =  | 0   |
| Multiple dependent claim(s) (if applicable)   |   | +\$270.00 (968)   |
| <b>TOTAL OF ABOVE CALCULATIONS =</b>  |   | \$ 1,000.00   |
| Reduction for 1/2 for filing by small entity, if applicable (see below).  |   | \$ 500.00 -   |
| <b>SUBTOTAL =</b>   |   | \$ 500.00   |
| Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)).  |   | 20 <input type="checkbox"/> 30 <input type="checkbox"/> \$ -- |
| <b>TOTAL NATIONAL FEE =</b>   |   | \$ 500.00   |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property +  |   | \$ --   |
| <b>TOTAL FEES ENCLOSED =</b>  |   | \$ 500.00   |
|   |   | Amount to be:<br>refunded \$                                  |
|   |   | charged \$  |
| a. <input checked="" type="checkbox"/> Small entity status is hereby claimed.   |   |   |
| b. <input checked="" type="checkbox"/> A check in the amount of \$ 500.00 to cover the above fees is enclosed.  |   |   |
| c. <input type="checkbox"/> Please charge my Deposit Account No. 02-4800 in the amount of \$ to cover the above fees. A duplicate copy of this sheet is enclosed.   |   |   |
| d. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed. |   |   |
| NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.                               |   |   |
| SEND ALL CORRESPONDENCE TO:   |   |   |
| Benton S. Duffett, Jr.<br>BURNS, DOANE, SWECKER & MATHIS, L.L.P.<br>P.O. Box 1404<br>Alexandria, Virginia 22313-1404<br>(703) 836-6620  |   |   |
| <br>SIGNATURE   |   |   |
| <u>Benton S. Duffett, Jr.</u><br>NAME   |   |   |
| <u>22,030</u><br>REGISTRATION NUMBER  |   |   |

09/937851  
4:0 Rec'd PCT/PTO 28 SEP 2001

Patent  
Attorney's Docket No. 003300-852

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of )  
                              )  
                              )  
SHOAA ABDUL RAHMAN et al. ) Group Art Unit: (unassigned)  
                              )  
                              )  
Application No.: (unassigned) ) Examiner: (unassigned)  
                              )  
                              )  
Filed: September 28, 2001 )  
                              )  
                              )  
For:   A PROCESS FOR STERILIZING A )  
      BIOLOGICALLY CONTAMINATED )  
      ENCLOSURE                )

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

This is a national phase application of International Application No.  
PCT/SE00/00597, filed March 28, 2001.

Please amend the Application as indicated.

**IN THE ABSTRACT:**

Please add the Abstract of the Disclosure that is provided on a separate sheet.

**IN THE CLAIMS:**

Kindly replace Claims 1, 3, 5 and 6 as follows:

1. (Amended) A process for sterilizing a biologically contaminated enclosure  
in which the enclosure is brought into contact with formaldehyde, said formaldehyde being  
released from formalin by heat generated by an exothermic reaction occurring in the

presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing formaldehyde from said formalin, wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, and said second reagent (B) comprising a peroxide compound or a precursor thereof.

3. (Amended) A process of claim 1, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin there are 180 to 210 grams of hexamethylenetetramine.

5. (Amended) A process of claim 1, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

6. (Amended) A process of claim 1, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

Please add the following new Claims 7 to 20:

7. (New) A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing

formaldehyde from said formalin, wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, in admixture with sulphur sublime, red iron oxide, silica and citric acid, and said second reagent (B) comprising a peroxide compound or a precursor thereof.

8. (New) A process of claim 2, wherein said reagent (B) comprises a hydrogen peroxide solution.

9. (New) A process of claim 2, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin 180 to 210 grams of hexamethylenetetramine.

10. (New) A process of claim 7, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin the following ranges of constituents of reagent (A) are utilized: up to 10 grams of sulphur sublime, up to 10 grams of red iron oxide, up to 5 grams silica, up to 5 grams of citric acid, and 180 to 210 grams of hexamethylenetetramine.

11. (New) A process of claim 2, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

12. (New) A process of claim 3, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

13. (New) A process of claim 4, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

14. (New) A process of claim 7, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

15. (New) A process of claim 8, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

16. (New) A process of claim 9, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

17. (New) A process of claim 2, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

18. (New) A process of claim 7, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

19. (New) A process of claim 8, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

20. (New) A process of claim 9, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

Application No. (unassigned)  
Attorney's Docket No. 003300-852  
Page 6

**REMARKS**

The present Amendment modifies the claim format and eliminates the use of multiple dependency.

The examination and allowance of the Application respectfully is requested.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: Benton S. Duffett Jr.  
Benton S. Duffett, Jr.  
Registration No. 22,030

P.O. Box 1404  
Alexandria, Virginia 22313-1404  
(703) 836-6620

Date: September 28, 2001

**Attachment to Preliminary Amendment dated September 28, 2001**

**Marked-up Claims 1, 3, 5 and 6**

1. (Amended) A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing formaldehyde from said formalin, [characterized in that] wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, [optionally in admixture with sulphur sublime, red iron oxide, silica and citric acid,] and said second reagent (B) comprising a peroxide compound or a precursor thereof.

3. (Amended) A process of [any one of claims 1-2] claim 1, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin [the following ranges of constituents of reagent (A) are needed: (0-10) grams of sulphur sublime, (0-10) grams of red iron oxide, (0-5) grams silica, (0-5) grams of citric acid and (180-210)] there are 180 to 210 grams of hexamethylenetetramine.

5. (Amended) A process of [any one of claims 1-4] claim 1, wherein the ratio between the reagents (A, B) is [0.7-1.5] 0.7 to 1.5.

**Attachment to Preliminary Amendment dated September 28, 2001**

**Marked-up Claims 1, 3, 5 and 6**

6. (Amended) A process of [any one of claims 1-5] claim 1, wherein the formalin has a concentration of [10-40] 10 to 40% formaldehyde gas.

1

A PROCESS FOR STERILIZING A BIOLOGICALLY  
CONTAMINATED ENCLOSURE

The present invention relates to a process for sterilizing a biologically contaminated enclosure and particularly to a safe and easy chemical process for a well-controlled release or fumigation of formaldehyde gas from formalin. The process is intended for sterilizing biologically contaminated enclosures such as animal houses, hatcheries, feed stores, feed bins, feed tanks, feed mills, hospitals, medical instruments or other hard to reach areas in which the slowly and well controlled release of formaldehyde gas from easily available formalin solution is responsible for the complete sterilization by maximizing the excellent killing effects against bacteria, viruses and fungi.

15 BACKGROUND OF THE INVENTION

Discussion of related art

It is well known that formaldehyde gas is the mostly used gas in the sterilization of livestock buildings. It is an effective disinfectant against vegetative bacteria, fungi, spores and viruses if an adequate time of exposure and not less than 70% humidity are provided (Remington page 1171, Cooper & Mason 1964).

It can be easily absorbed by surfaces in both its gaseous or liquid state, as it has strong penetration power which increases in completely closed spaces.

In a study (Sykes, 1972) it was proved that formaldehyde can attain complete sterilization to a well closed room within two hours at 70% relative humidity.

Its mode of action on living cells is by reaction with the cellular protein and (DNA, RNA) amino acids (Russel, 1976).

Formalin, as a commercially available product, is an aqueous solution containing up to 37% by weight of formaldehyde, HCHO (30.03), with methanol added to prevent

polymerization. The solution is extensively used for disinfecting rooms, which have been subjected to infection, by:

5      1. Reaction of formalin with half its weight of potassium permanganate.

2. Spraying it on sheets hung in the room.

10     3. Releasing formaldehyde vapor from formalin into the room by a heat generator or heater.

4. Subliming paraformaldehyde powder at 218°C using a heater.

15     There are disclosed in WO 97/23247 Al antiperspirant pads which are impregnated with hexamethylenetetramine in an acidic or neutral medium for releasing formaldehyde.

From STN International, File Caplus, Accession no. 20 1985:492906, Document no. 103:92906, RO 85306 B, it is known to disinfect technological spaces by using formaldehyde which is rapidly released from a formalin solution by a strong exothermic reaction due to the addition of KMnO<sub>4</sub>.

25     In US patent no. 4356179 formaldehyde products are used as agricultural fungicides. The products can for example be obtained by adding hexamethylenetetramine to formaldehyde.

30     All the above mentioned methods have many disadvantages. Using potassium permanganate to evaporate formaldehyde results in a very vigorous and dangerous reaction, which is completed within few seconds, and this does not give enough time for the worker to escape safely and many fatal accidents have been reported in real life.

35     It can be proved that spraying formalin on sheets is non-reproducible and does not release enough formaldehyde gas as it is temperature-dependent as can be seen from the following table

| <u>Concentration and type of formaldehyde</u> | <u>Time needed to kill spores</u> | <u>Time needed to kill bacteria</u> |
|---|-----------------------------------|-------------------------------------|
| Formaldehyde gas                              | 2 hours                           | 2 hours                             |
| 8% formalin                                   | 18 hours                          | 18 hours                            |
| 0.5% formalin                                 | 2-4 days                          | 6-12 hours                          |

Formaldehyde gas is thus described as being a sterilizing agent, while formalin (10% strength) is described as being an antiseptic agent; hence there is a  
 5 need to use the formaldehyde gas as a sterilizer rather than using the formalin, as formalin is time consuming and requires diligent application through decontamination.

Reference: Remington's Pharmaceutical Sciences, 18th.  
 10 Ed., pages 1171, 1470. Merck Index 11th Ed.

Further, contaminated, highly sophisticated electronic and dental equipment may be damaged by sterilizing liquid agents like those described in US Patent 31779 or by heat or autoclaving etc. US Patent 5552112 introduced  
 15 a new method for sterilizing a metallic surgical instrument with microwave radiation but the method suffers from non-availability of microwave source to everyone and high cost. As mentioned in US Patent 5552112, gas sterilization with an ethylene oxide mixture is acceptable for  
 20 both hanpieces and burs. However, this is impractical because of cost of equipment, long sterilization and aeration times involved, and cost of providing adequate protection for personnel. Alkaline glutaraldehyde (2%) as mentioned by Boucher in US Patent 3912450, is used to  
 25 sterilize equipment, but it must be used for 10 hours to kill spore-forming organisms or tuberculosis microorganisms and is irritating to tissue.

Many workers could successfully release formaldehyde gas using generators or heaters and some patents have been  
 30 published like US Patents: 4585624, 665794, 1837264, 2993832, 3694146, 3816074, 3898038 and 4166087. All these patents describe different apparatus systems for vaporiz-

ing formaldehyde and dispensing it into the enclosure to be sterilized for contact with contaminants. However, such methods suffer from high cost of instruments, maintenance, availability, complexity and restricted volume of formalin to be evaporated. No work has ever been known to use a safe chemical process for the evaporation of formaldehyde as an alternative for the unsafe usage of potassium permanganate or other conventional methods.

10 **SUMMARY OF THE INVENTION**

The present invention provides an ideal, chemical and safe process for releasing formaldehyde gas from a formalin solution by evaporation using heat generated by an exothermic reaction in order to simplify and optimize the 15 use of formaldehyde gas in sterilizing closed spaces, like houses, buildings for housing animals, hospitals, operating rooms, stores, hotels, bath rooms or any object needing to be sterilized.

It is therefore an object of the present invention 20 to provide a process for sterilizing enclosures, such as animal houses, hatcheries, feed stores, feed bins, feed tanks, feed hauling truck bins or tanks, feed mills or other hard to reach areas, or rooms in hospitals, clinics, research laboratories and the like by chemically releasing gaseous formaldehyde into the enclosure for contact with contaminants.

The present invention is based on the finding that 30 the release of formaldehyde gas can be easily provided by heat generated by an exothermic reaction occurring in the presence of formalin. The released formaldehyde gas can be used for sterilizing closed spaces of the kind exemplified above. Exothermic chemical reactions involving various reagents are known to one skilled in the chemical art. In general, a chemist can chose any reagents for 35 such exothermic reactions. Needless to say, it is advantageous to chose reagents, which are easily available and cheap. Once chosen the amounts of reagents sufficient to

generate heat for releasing gaseous formaldehyde for sterilization from formalin are easily determined by one skilled in the art. As an example of appropriate reagents there can be mentioned hexamethylenetetramine and peroxide compounds or precursors of peroxide compounds, which when mixed, and in the presence of formalin, bring about an exothermic reaction releasing formaldehyde gas for sterilizing.

According to the invention the exothermic reaction is provided by addition of reagents to the formalin in amounts sufficient to generate heat for releasing formaldehyde from the formalin. In accordance therewith, the reagents comprise a first reagent A and a second reagent B, said reagent A comprising hexamethylenetetramine, optionally in admixture with sulphur sublime, red iron oxide, silica, preferably that sold under the tradename Aerosil, and citric acid, and the second reagent B comprising a peroxide compound, such as hydrogen peroxide, or a precursor thereof.

According to a most preferred embodiment, the reagent A comprises Methenamine (hexamethylenetetramine) and the reagent B comprises a hydrogen peroxide solution. Mostly preferred, said solution contains 10-50% hydrogen peroxide.

In accordance with the present invention the formalin, for example having a concentration of 10-40% formaldehyde gas, is mixed with the reagent A comprising hexamethylenetetramine and the reagent B, for example hydrogen peroxide. As explained below, the temperature of the reaction solution will increase spontaneously by the exothermic chemical reaction and the production of formaldehyde gas starts effectively at 60°C. The temperature rises and release of formaldehyde gas reaches the maximum value at 90°C.

As non-limiting examples of peroxide compounds the following can be mentioned: ammonium peroxosulfate, potassium peroxodisulfate, hydrogen peroxide, acetyl pero-

ide, benzoyl peroxide and cumene hydroperoxide.

According to a most preferred embodiment, wherein said reagents comprise hexamethylenetetramine and hydrogen peroxide the ratio between these is within the range 5 of 0.7-1.5.

DETAILED DESCRIPTION OF THE INVENTION:

The present invention is illustrated by the following example:

10 To evaporate 10 ml up to maximum 3500 ml (preferably 2000 ml) of formalin containing 10%-40% of formaldehyde gas in water:

15 The above formalin quantity is mixed with 100g-400g, preferably 200 g powder (A) which consists of five ingredients as follows:

| Ingredient      | Range in grams | Preferably in grams |
|-----------------|----------------|---------------------|
| Sulphur sublime | 0-10           | 0.30                |
| Iron oxide, red | 0-10           | 0.30                |
| "Aerosil"       | 0-5            | 0.40                |
| Citric acid     | 0-5            | 4.00                |
| Methenamine     | 180-210        | 195.00              |

20 In case of sterilizing highly sophisticated medical or dental instruments or other similar objects Methenamine (hexamethylenetetramine) without the rest of chemicals shown in the above table should be used.

After mixing the above quantity of powder (A) with the above amount of formalin, (100ml-400ml, best results with 200ml) liquid (B) is added.

25 It is the powder part, which controls the reaction, and provides enough time for the operator before the onset of the evaporation process.

Liquid (B) is hydrogen peroxide having a concentration of 10%-50% (best results with 50%). It is the heat 30 generated by mixing powder (A) and liquid (B) which helps

in heating and evaporation or controlled fumigation of formalin.

After about five minutes, the temperature of the solution increases spontaneously by the exothermic chemical reaction and the production of formaldehyde gas starts effectively at 60° C. With the rise of the temperature of the exothermic reaction the evaporation of formaldehyde gas becomes very strong and reaches the maximum at 90° C (after about 10 minutes).

10

EXAMPLES:

Example 1:

In order to illustrate the invention the following non-limiting example is given:

A 4000 m<sup>3</sup> (10000 bird capacity) chicken house was chosen to be sterilized immediately after the complete evacuation of the building and before the new bird cycle. The house was left as such without further cleaning. Swabs were taken from different representative areas of the house (ceiling, flour etc.). All windows and openings were tightly closed. The house was humidified with water to obtain at least 70% relative humidity. The chosen formalin amount for the experiment was 2 lit per 100m<sup>3</sup>, so 40 lit of (37%) formaldehyde solution were divided into 20 plastic containers (10 lit capacity each) such that each container contains 2 lit of formalin. The containers were placed evenly in the house. To each container 200 g of powder (A) were added and consisting of:

| <u>Ingredient</u> | <u>Quantity (g)</u> |
|-------------------|---------------------|
| Sulfur Sublime    | 0.30                |
| Iron oxide, red   | 0.30                |
| "Aerosil"         | 0.40                |
| Citric acid       | 4.00                |
| Methenamine       | 195.00              |
| TOTAL             | 200.00              |

Powder (A) was mixed thoroughly with formalin and thereafter 200 ml of liquid (B) (50% hydrogen peroxide) were added to every container and mixed.

In every container solution temperatures were recorded against time, and the following average solution temperatures were obtained against time.

| <u>Time in minutes</u> | <u>Solution Temperature °C</u> |
|------------------------|--------------------------------|
| 3                      | 40                             |
| 5                      | 60                             |
| 7                      | 80                             |
| 10                     | 95                             |
| 30                     | 95                             |
| 40                     | 85                             |
| 50                     | 75                             |
| 60                     | 65                             |
| 100                    | 40                             |

Formaldehyde started evaporating "effectively" when the temperature of the solution reached 60° C after 5 minutes of mixing all ingredients together.

After 10 minutes the evaporation became extensive (boiling).

This temperature was attained for 20 minutes before it declined and reached room temperature after two hours.

The chicken house was kept closed and empty for 48 hours after which it was well ventilated and swabs were taken again randomly and analyzed for total fungal and bacterial count.

| <u>Average count before sterilization</u> | <u>Average count after sterilization</u> |
|---|--|
| 1000,000 colonies/g                       | 2 colonies/g                             |

#### 20 Example 2:

Calculate the size of the house and then put in separate containers (20 liters capacity) two liters of Formalin for each 100 cubic metre. Add 200 g of reagent A by mixing with the standard Formalin in all containers; steer the mix after adding the powder until it is com-

pletely dissolved, then add 200 ml of Reagent B to the mixture and leave the house. After five minutes evaporation starts and the release of formaldehyde gas continues for more than four hours and it is preferable to keep the house closed overnight. The production of formaldehyde gas starts effectively at 60°C. The evaporation becomes very strong and reaches the maximum at 90°C after 10 minutes. Fumigation works best at temperatures above 18°C, therefore the house temperature should be maintained above this level and the humidity should be around 70%.  
The composition of reagent A is sulphur, iron oxide, di-calcium phosphate and hexamethylenetetramine. Reagent A controls the formaline evaporation reaction and provides a suitable time before the onset of evaporation. Reagent B is a liquid comprising 50% hydrogen peroxide which is sufficient to evaporate two litres of "37% formalin solution. Reagent B assists in heating and functions as a catalyst for evaporation of formalin.

Reagent A is to be kept in a cool place at a temperature below 30°C, out of reach of children and feed stock places. Reagent B is also to be kept in a cool place at a temperature below 30°C, out of reach of children and feed stock places.

Example 3:

The procedure of this example was carried out in the same manner as in example 2 with the exception of that the composition of reagent A is 100% hexamethylenetetramine powder. The results of this experiment were also excellent as in the aforementioned experiments.

The present invention, therefore, is well suited and adapted to attain the intended objects and has the advantages and features mentioned as well as others inherent therein. The foregoing description is provided to illustrate the invention, and is not to be construed as a limitation.

REFERENCES

- 5 1. Russell, A.D. 1976 . Inactivation of non-sporing bacteria by gases. SOC.APPL.Bacterial., 5, 61-68.
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## CLAIMS

1. A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing formaldehyde from said formalin, characterized in that said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, optionally in admixture with sulphur sublimate, red iron oxide, silica and citric acid, and said second reagent (B) comprising a peroxide compound or a precursor thereof.

2. A process of claim 1, wherein said reagent (B) comprises a hydrogen peroxide solution.

3. A process of any one of claims 1-2, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin the following ranges of constituents of reagent (A) are needed: (0-10) grams of sulphur sublimate, (0-10) grams of red iron oxide, (0-5) grams of silica, (0-5) grams of citric acid and (180-210) grams of hexamethylenetetramine.

4. A process of claim 2, wherein the solution contains 10-50% hydrogen peroxide.

5. A process of any one of claims 1-4, wherein the ratio between the reagents (A, B) is 0.7-1.5.

6. A process of any one of claims 1-5, wherein the formalin has a concentration of 10-40% formaldehyde gas.

**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY  
(Includes Reference to Provisional and PCT International Applications)**

Attorney's Docket No.  
003300-852

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;  
I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**A PROCESS FOR STERILIZING A BIOLOGICALLY CONTAMINATED ENCLOSURE**

the specification of which (check only one item below):

is attached hereto.

was filed as United States application

Number \_\_\_\_\_

on \_\_\_\_\_

and was amended

on \_\_\_\_\_ (if applicable).

was filed as PCT international application

Number \_\_\_\_\_

on \_\_\_\_\_

and was amended

on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(e) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

**PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §119:**

| COUNTRY<br>(if PCT, indicate "PCT") | APPLICATION NUMBER | DATE OF FILING<br>(day, month, year) | PRIORITY CLAIMED<br>UNDER 35 U.S.C. §119                            |
|-------------------------------------|--------------------|--------------------------------------|---|
| Sweden                              | 9901137-1          | 29 March 1999                        | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
|                                     |                    |                                      | <input type="checkbox"/> Yes <input type="checkbox"/> No            |
|                                     |                    |                                      | <input type="checkbox"/> Yes <input type="checkbox"/> No            |
|                                     |                    |                                      | <input type="checkbox"/> Yes <input type="checkbox"/> No            |

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONTINUED)**  
 (Includes Reference to Provisional and PCT International Applications)

Attorney's Docket No.

003300-852

I hereby claim the benefit under Title 35, United States Code, §120 of any United States applications(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose to the Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations §1.56, which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

**PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:**

| U.S. APPLICATIONS       |                  | STATUS (check one) |         |           |
|-------------------------|------------------|--------------------|---------|-----------|
| U.S. APPLICATION NUMBER | U.S. FILING DATE | PATENTED           | PENDING | ABANDONED |
|                         |                  |                    |         |           |
|                         |                  |                    |         |           |
|                         |                  |                    |         |           |

| PCT APPLICATIONS DESIGNATING THE U.S. |                 |  |
|---------------------------------------|-----------------|--|
| PCT APPLICATION NO.                   | PCT FILING DATE | U.S. APPLICATION NUMBERS ASSIGNED (if any) |
| SE00/00597                            | 28 March 2000   |  |
|                                       |                 |  |

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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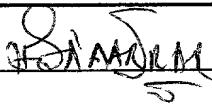
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONTINUED)  
(Includes Reference to Provisional and PCT International Applications)ATTORNEY'S DOCKET NO.  
003300-852

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